Characterizing a Novel Low-Voltage Pulsed EB: Challenges in Dosimetry

By K. Benjamin Richter, Alon V. McCormick, L. E. Scriven and Douglas E. Weiss low-voltage pulsed electron beam has been constructed that demonstrates energy efficiency and enhances the overall polymerization of acrylic monomers when compared to traditional, continuous low-voltage electron beams. The novelty of this equipment and the need to evaluate different configurations and explore operating ranges required frequent and cost-effective dosimetry. Two types of dosimetry were used: electronic dosimetry, relying on the

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> short duration of the electron bursts; and chemical dosimetry, evaluated with the free public domain image processing tool ImageJ.

Introduction

Low-voltage pulsed electron beams (PEB) convert acrylic monomers more quickly and completely than continuous low-voltage electron beams (EB) at equal doses, as Weiss and Dunn have shown.¹ PEB exploits the inherent heterogeneous nature of energy deposition along electron tracks that compartmentalize free radical initiation sites, thereby suppressing second-order termination reactions in favor of firstorder propagation. The conversion kinetics in mono- and multi-functional acrylate systems have been used to benchmark PEB curing against continuous EB and UV curing. These acrylates were converted quickly and more completely by PEB.

In contrast to UV and conventional low-voltage EB curing, PEB offers lower overall energy demand, higher process speeds, and, in contrast to UV, obviates the need for photoinitiators. It is therefore possible, through appropriately timed pulses, to cure neat acrylic monomers in-line to produce desired coatings more efficiently and at speeds required in industrial processes. These experimental results can be applied directly to industrial web widths, and equipment design can be readily extended to large facilities.

The observed changes in properties of these acrylates with changes in operating parameters are consistent with predictions from a kinetic model of PEB curing, which is built on the analysis by Wen and McCormick of the kinetics of pulsed UV curing that includes the fall of species mobility with rising conversion.² This PEB model also illustrates the benefits of low-voltage PEB.

To reduce overall energy demand, improve process speeds, and obviate the need for photoinitiators, PEB curing may thus offer advantages over other radiation-curing methods for many future applications.

A New Electron Beam Apparatus

Weiss and Dunn's studies were performed on a prototype pulsed,

low-voltage electron beam apparatus that no longer exists. To confirm these results and evaluate the process, a new, more robust and flexible pulsed electron system was needed.3 A low-voltage PEB apparatus (Figure 1) was developed in cooperation with North Star Power Engineering, a division of Ionatron Inc. of Tucson, Ariz. The new system uses proprietary technology developed at North Star Power Engineering and is unique in its ability to uniformly expose an inerted, temperature-controlled 200 cm² sample chamber at atmospheric pressure with 80-160 keV electron energy in pulses of 5-20 µsec duration at frequencies of up to 2 kHz. The flexibility of this configuration is the result of a custom-built electron acceleration chamber, vacuum system, protective lead shielding, transformer, and computer-controlled pulse module. This configuration allows for a thorough and multi-faceted investigation of pulsed EB curing in the laboratory and may be scaled up to handle common industrial web widths needed for commercial applications.

Special Challenges

As with any new experimental setup, characterization of the beam's output had to precede any materials experimentation and be done in a cost-effective manner. The novelty of the equipment required frequent dosimetry tests at different configurations and operating ranges. An instrument-specific operating window defining the range of operating parameters was developed that established attainable voltages and dose, as were determined by the transient behavior and power consumption of the system during a pulse. Such an operating window proved especially useful in evaluating applications since the current PEB design may be scaled up to handle common web widths needed in practice. The need for

frequent dosimetry called for a fast characterization method; some uncertainty in the measurement was acceptable as long as trends could be seen that identified the effects of changing various operating parameters.

On the other hand, operating conditions need to be verified during each run by dosimetry to ensure proper irradiation conditions. Some type of chemical dosimetry is the industry standard. However, most systems are slow and equipment for evaluation can be costly. Longer waiting times to obtain dose measurements are acceptable if the approximate operating conditions are known.

Two dosimetry methods were used: first, a fast, electronic dosimetry system based on charge collectors that can give a rough but instant dose map of the sample chamber; second, a low-cost chemical dose mapping approach using free open-source software.

Electronic Dosimetry

The electronic dosimetry method relies on the short duration of the electron bursts from the PEB, which are simply measured by placing a charge collector of known capacitance in the beam's path. The energy delivered to the charge collector can then be determined by measuring the voltages of the beam and charge collector. By the definition of capacitance, the charge collected on a capacitor (Q) equals the voltage on the capacitor (V_e) times its capacitance (C), that is $Q=CV_e$.

The total energy per pulse (U)is simply the charge collected during the pulse times the energy with which this charge arrives, the operating voltage of the beam $(V_b): U=QV_b$.

Thus, the total absorbed energy in the electronic dosimeter per pulse is $U=CV_{e}V_{e}$, where C is known and

FIGURE 1

A new, custom-built pulsed electron beam apparatus



 V_c and V_b are both measured using an oscilloscope. In this manner, if the charge collector's signal is initially calibrated against a radiochromic chemical dosimeter, the inferred dose per pulse can be continuously monitored.

Several such charge collectors can be assembled into an array (Figure 2). Here an array of dimes was placed as charge collectors, each connected via a wire to a circuit board, from which voltages can be read manually. This arrangement provided for a convenient way to collect frequent dose readings at different regions of the sample chamber and could be automated with data acquisition software such as NI LabVIEW.

This is an easy-to-use method to monitor the electron beam's output in real time. When readings from all dimes in the array are combined and linearly interpolated, one can even construct a rough dose map of the entire sample chamber (Figure 3). Comparison with a chemical dose map (Figure 4) shows that while the electronic dose map is somewhat crude, it gives a good first approximation of a photochemical dosimeter system.

Chemical Dosimetry

The dose map of Figure 4 is the result of chemical dosimetry. The dosimeter used, the B3 radiochromic film system, takes considerable time to measure, but is more easily calibrated than the electronic dosimetry mentioned earlier. The film has to be irradiated and heated in an oven for several minutes to let the full color develop.⁴ The traditional way to evaluate the film is to measure the optical absorbance at a specified wavelength using a spectrophotometer. This approach, however, only allows for spot measurements of delivered dose.

Alternatively, the developed film can be scanned in a flatbed scanner and the resulting image evaluated digitally.⁵ The advantage then is that large areas of film can be evaluated and it is possible to construct detailed dose maps, which have resolutions only limited by the resolution of the flatbed scanner. This can be done with the open-source image processing and analysis tool ImageJ that is made available by the National Institutes of Health.⁶ The radiochromic dosimeters were calibrated against known doses from a wellcharacterized continuous electron beam that is in operation at 3M Company.

Sample films can be scanned on a flatbed scanner that allows for scanning without color correction. To obtain a full dose map (Figure 4), the following manipulations were needed, all of which can be performed in ImageJ:

- Split the image into the three color channels red, green, and blue and discard all but green;
- Invert the image;
- Measure pixel values for the calibration standards and assign doses to each; and
- Calibrate the image with a third-degree polynomial to these standards.

ImageJ will display the total delivered dose rather than pixel

FIGURE 2

Dimes are convenient, low-cost charge collectors that can be assembled in an array



values anywhere in the scanned image. Figure 4 was created by selecting the area of interest on a large sheet of dosimeter film and creating a surface plot. Notice how the dimensions of the selected region are indicated on the plot and how the values are given in kGy, following the calibration input. ImageJ is fully scriptable, and all of the above operations can be combined as a plug-in to the program that allows for the full-image manipulation in a single step.

Conclusion

Despite a large and growing user base, there are few convenient approaches to low-voltage electron beam dosimetry. As EB-curing research gains momentum in academia and industry alike, much can be gained from developing new methods and rethinking

FIGURE 3



FIGURE 4



older ones. Approaches to EB dosimetry should be inexpensive and easy-to-use, but not simplistic. Two relatively inexpensive approaches to low-voltage EB dosimetry have been presented:

- A fast, low-precision electronic dosimetry.
- A chemical dosimetry system.

These systems may offer significant cost savings. Greater end-user involvement will foster better understanding of the EB process, which in turn may lead to more rapid development of new EB technologies.

References

- D. E. Weiss and D. S. Dunn. Pulsed polymerization of acrylate monomers using electron beam. Conditions for achieving single-phase heterogeneous polymerization. Radiation Physics and Chemistry, 65:281-288, 2002.
- M. Wen and A. V. McCormick. A kinetic model for radical trapping in photopoly-merization of multifunctional monomers. Macromolecules, 33:9247-9254, 2000.

- D. E. Weiss, D. S. Dunn, K. B. Richter, and R. Adler. Pulsed electron beam polymerization. In Pressure Sensitive Tape Council TECH XXVIII Global Conference Proceedings, Baltimore, May 4-6, 2005.
- 4. GEX Corporation. B3 Film Dosimeters-Technical Usage Information, 2006.
- J. Helt-Hansen and A. Miller. RisøScan—a new dosimetry software. Radiation Physics and Chemistry, 71:359-362, 2004.
- M. D. Abràmoff, P. J. Magelhaes, and S. J. Ram. Image processing with ImageJ. Biophotonics International, 2004(7): 36-42, July 2004.

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